

In re Application of:  
Cherkasov et al.

Filed: June 22, 2005

For: Late Transition Metal Olefin  
Polymerization and  
Oligomerization Catalysts And  
Related Preparation Methods

§  
§ Examiner: Caixia Lu  
§  
§ Group Art Unit: 1713  
§  
§ Docket No: 2003B073  
§  
§ Confirmation No.: 1152  
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§  
§ Date: September 15, 2006

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

I, Jo Ann M. Canich, declare as follows:

1. I am one of the inventors of the subject application and one of the authors of the attached Exhibit A.
2. Exhibit A is a copy of a patent memorandum that I and others submitted to the ExxonMobil Chemical Company Law Technology Department as part of an invention disclosure that forms the basis of the present application. The patent memorandum indicates conception and reduction to practice of the claimed invention before June 25, 2002. All masked dates in Exhibit A are prior to June 25, 2002.
3. The subject matter of Exhibit A was diligently prepared and filed as U.S. Patent Application Serial No. 10/517,104 beginning at a time prior to June 25, 2002, until the filing of priority application U.S. Provisional Application Serial No. 60/396,370 filed on July 17, 2002.

4. Exhibit A shows that the invention which forms the subject matter of the pending claims in the above-captioned patent application was conceived in the United States, specifically in the State of Texas, before June 25, 2002, and diligently reduced to practice in the United States by at least July 17, 2002, the filing date of U.S. priority provisional application, Serial No. 60/396,370.

5. Exhibit A includes a description of transition metal compounds containing substituted and unsubstituted catecholate ligands, as recited in, for example, claim 1. More particularly, Exhibit A includes a description of a transition metal compound represented by the formula LMX wherein M is a Group 3 to 11 metal; L is a bulky bidentate or tridentate neutral ligand that is bonded to M by two or three heteroatoms and at least one heteroatom is nitrogen; and X is a substituted or unsubstituted catecholate ligand, as recited in claim 1. (*See, Exhibit A, pages 2-6.*)

6. Exhibit A is offered as supporting evidence that the compounds of the present invention were conceived in the United States before June 25, 2002, the earliest publication date of U.S. Patent No. 6,410,768, which published on June 25, 2002 to Llatas et al., and diligently reduced to practice by filing the priority application.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

9/15/06  
DATE

Jo Ann M. Canich  
Jo Ann M. Canich, Ph. D.

# PATENT MEMORANDUM

ExxonMobil Chemical Company

INVENTOR(S) V. K. Cherkasov, M. P. Bubnov, Baiyi Zhao, Kevin R. Squire, Smita Kacker and Jo Ann M. Canich		MEMO IN DATE 1991
DESCRIPTIVE TITLE New diamagnetic Ni, Co and Fe olefin polymerization and oligomerization catalysts and the preparation thereof.		
BUSINESS UNIT Polymer Science		BRIEF ABSTRACT OF INVENTION New diamagnetic late transition metal pre-catalysts that are capable of polymerizing olefins have been discovered. Because these complexes are diamagnetic and typically soluble in common organic solvents, these pre-catalysts have an advantage over typical paramagnetic late transition metal pre-catalysts in terms of ease of synthesis, characterization and use in polymerization reactions.
STATUS OF INVENTION (EXISTING OR CONTEMPLATED EXPERIMENTAL, COMMERCIAL OR PLANT SCALE USE).  Existing		<div style="font-size: 2em; font-weight: bold; text-align: center;">DATE</div> <div style="font-size: 2em; font-weight: bold; text-align: center;">DATE</div>
WERE YOU WORKING FULL OR PART TIME ON ANY GOVERNMENT CONTRACT AT, OR WITHIN ONE YEAR PRIOR TO THE TIME THE INVENTION WAS FIRST CONCEIVED OR TESTED?  <input type="checkbox"/> YES <input type="checkbox"/> NO CONTRACT NO.(S):		
FIRST WRITTEN DESCRIPTION?	HEREWITH OR FILED ON DATE:	DATE OF FIRST TEST
DATE	TO WHOM AND WHERE	WHERE RECORDED? (E.G. NOTEBOOK & PAGE)
COMMUNICATION TO OTHERS?		21503-040-001
DATE		

With the discoveries of Brookhart, Gibson, and others, the new frontier in olefin polymerization catalysis is now focussed on late transition metal chemistry. The catalyst precursors of this type are typically nickel, cobalt or iron dihalide compounds complexed with bidentate or tridentate chelating ligands. These catalyst precursors are typically tetrahedral paramagnetic complexes that are difficult to characterize and purify. Characterization techniques are generally limited to IR, elemental analysis and x-ray crystallography. We have discovered that by replacing the two halide ligands with one chelating catecholate ligand that these new complexes can be stabilized into a square planar geometry that provides a diamagnetic complex that can be readily characterized by NMR spectroscopy. An additional benefit is that the compounds are generally more readily soluble in common organic solvents. This is beneficial for use in continuous reactors or in preparing supported catalysts. This chemistry should also be extendable to other chelating ligands such as but not limited to substituted or unsubstituted conjugated dienes, substituted or unsubstituted chelating divalent hydrocarbyl ligands, substituted allyl ligands, and other chelating ligands that will stabilize the pre-catalyst in a square planar geometry or pseudo square-planar geometry.

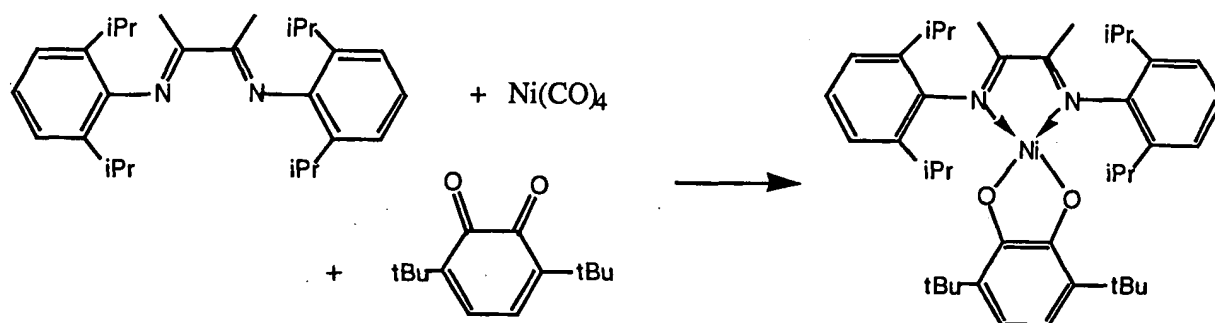
The catalysts of this invention are capable of producing linear or branched polymers of high or low molecular. Polymer architecture is dependent on the ligand geometry about the metal center. Some catalysts of this invention are also capable of producing alpha olefin from ethylene.

The new complexes of this type can be synthesized by at least three different methods.

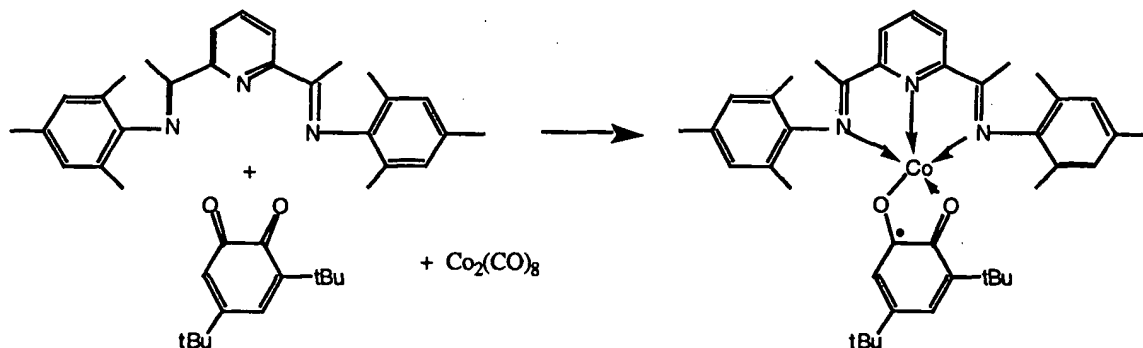
The first method of preparation uses a metal carbonyl complex, a bidentate or tridentate chelating ligand and a 1,2-benzoquinone complex to form the desired complex. Depending on the metal complex used, the oxidation of the metal may be a one or two electron oxidation. Examples of this redox reaction is illustrated below:

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SIGNATURE (FIRST NAME IN FULL) <i>Smita Kacker</i> (EMRE, CSR)	SIGNATURE (FIRST NAME IN FULL) <i>Jo Ann M. Canich</i>	SIGNATURE (FIRST NAME IN FULL) <i>Kevin R. Squire</i>
SIGNATURE (FIRST NAME IN FULL) <i>Baiyi Zhao</i>	SIGNATURE (FIRST NAME IN FULL)	SIGNATURE (FIRST NAME IN FULL)
WITNESSED, READ & UNDERSTOOD BY <i>Walter M. ...</i>	WITNESSED, READ & UNDERSTOOD BY	WITNESSED, READ & UNDERSTOOD BY
INVENTOR EMPLOYMENT CONTRACT	INVENTOR EMPLOYMENT CONTRACT	INVENTOR EMPLOYMENT CONTRACT
ExxonMobil Chemical Co. DIVISION:	ExxonMobil Chemical Co. DIVISION:	ExxonMobil Chemical Co. DIVISION: <i>Polymer Science</i>
OTHER:	OTHER:	OTHER:

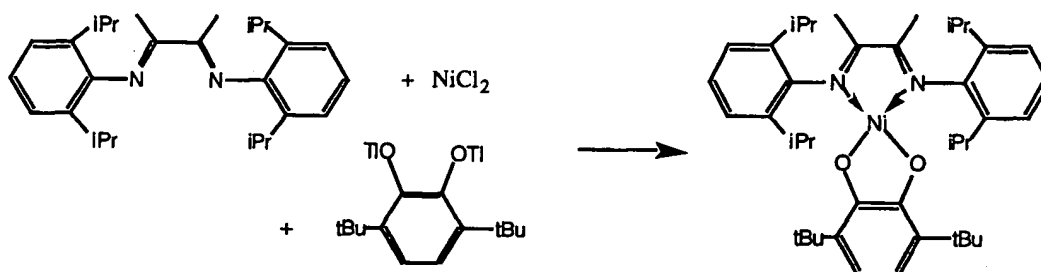
Formation of a nickel (II) complex:



Formation of a cobalt (I) complex:

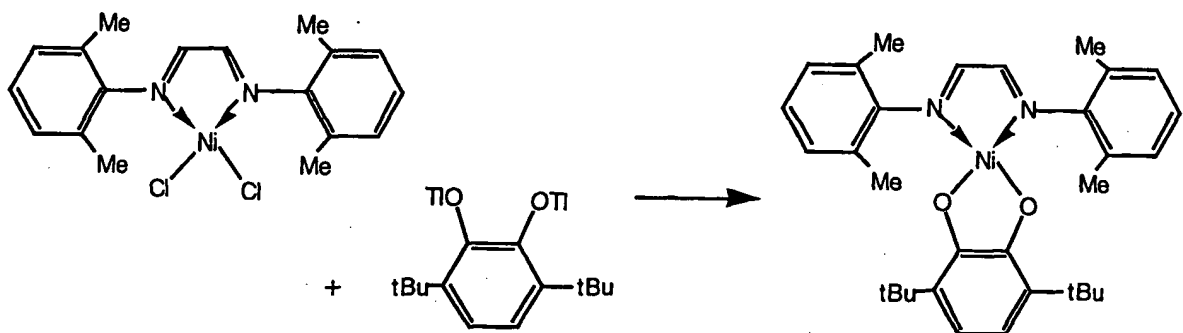


The second method of preparation uses a metal dihalide, a chelating ligand and a dithalium 1,2- catecholate complex or other salt complex to form the desired complex. An example of this reaction is illustrated below:

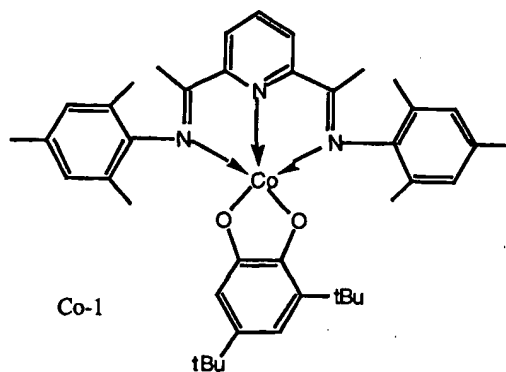
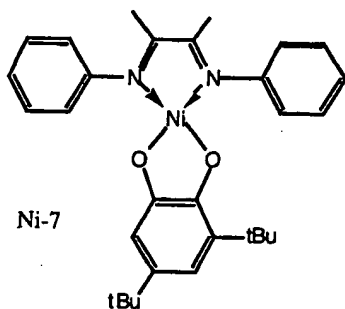
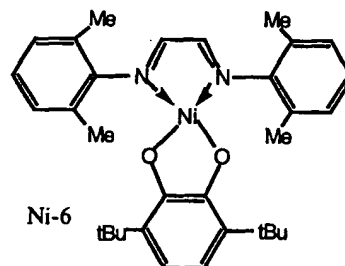
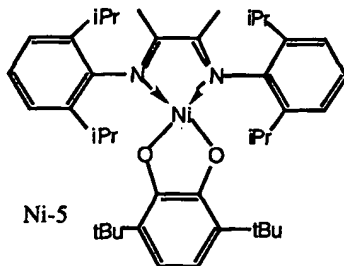
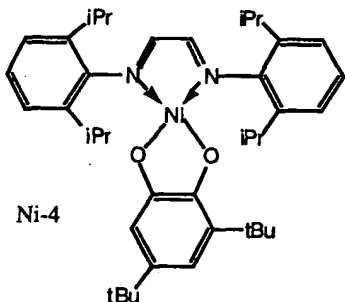
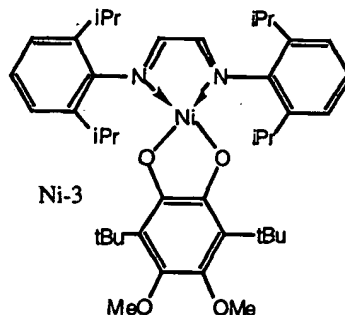
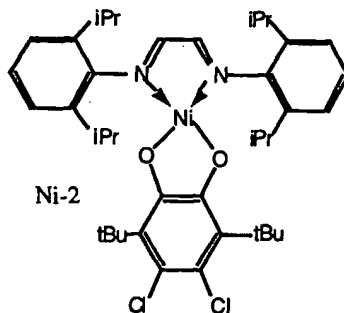
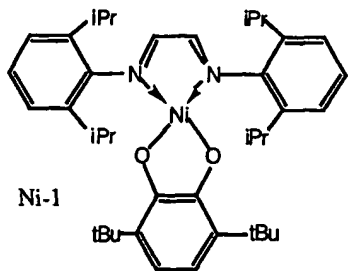


The third method of preparation requires the use of the chelated metal dihalide salt with a thallium 1,2- catecholate complex or other salt complex. This reaction is illustrated below:

INVENTOR(S)		DATE
SIGNATURE (FIRST NAME IN FULL)	SIGNATURE (FIRST NAME IN FULL)	
<i>Binita Kacker</i>	<i>Jim Canal</i>	
SIGNATURE (FIRST NAME IN FULL)	SIGNATURE (FIRST NAME IN FULL)	
<i>Basil</i>	<i>KR Squire</i>	
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<i>Walt</i>	<i>Ray</i>	
INVENTOR EMPLOYMENT CONTRACT	INVENTOR EMPLOYMENT CONTRACT	
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OTHER:	OTHER: <i>Polymer Science</i>	



The following compounds have been synthesized by one of the above methods:



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<i>Bmitakacker</i>	<i>J Mcand KR Squire</i>	
SIGNATURE (FIRST NAME IN FULL)	SIGNATURE (FIRST NAME IN FULL)	
<i>Borge Shao</i>		
WITNESSED, READ & UNDERSTOOD BY	WITNESSED, READ & UNDERSTOOD BY	
<i>Walter T. Natus</i>	<i>[Signature]</i>	
INVENTOR EMPLOYMENT CONTRACT	INVENTOR EMPLOYMENT CONTRACT	
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The above compounds when activated by a commonly known activator such as methyl alumoxane form active catalysts for the polymerization or oligomerization of olefins. Activators include alumoxanes such as methyl alumoxane, modified methylalumoxane, ethyl alumoxane, iso-butyl alumoxane and the like; aluminum alkyls such as trimethyl aluminum, triethyl aluminum, triisopropyl aluminum, triisobutyl aluminum and the like; Lewis acid activator used in combination with alumoxanes or aluminum alkyls such as triphenyl boron, tris-perfluorophenyl boron, tris-perfluorophenyl aluminum and the like; Ionic activators used in combination with alumoxanes or aluminum alkyls such as dimethylanilinium tetrakis perfluorophenyl borate, triphenyl carbonium tetrakis perfluorophenyl borate, dimethylanilinium tetrakis perfluorophenyl aluminate, and the like.

Additionally, the active catalyst component can be formed *in situ* by mixing together the bidentate or tridentate chelating ligand, the metal carbonyl, and the benzoquinone. This mixture is then added to the reactor and an activator is added, preferably in the presence of olefin. Other neutral metals or metal complexes may be used in place of the metal carbonyl, for example, in place of nickel carbonyl ( $\text{Ni}(\text{CO})_4$ ), bis(1,5-cyclooctadiene) nickel(0), bis(triphenylphosphine)nickel dicarbonyl, tetrakis(trifluorophosphine) nickel(0), nickel powder and the like; in place of cobalt carbonyl ( $\text{Co}_2(\text{CO})_8$ ), cobalt tricarbonyl nitrosyl, cobalt powder and the like; in place of iron carbonyl ( $\text{Fe}(\text{CO})_5$ ,  $\text{Fe}_3(\text{CO})_{12}$  or  $\text{Fe}_2(\text{CO})_9$ ), cyclohexadiene iron tricarbonyl, cyclooctatetrene iron tricarbonyl, or iron powder.

### Experimental

General synthetic procedure for compounds Ni-1, Ni-2, Ni-3, Ni-4 and Ni-5: Nickel tetracarbonyl (0.171 g, 1 mmol) was condensed into evacuated ampoule (approximately 200 ml volume) containing the corresponding benzoquinone (1 mmol) and diazabutadiene (1 mmol) in 20 ml of degassed toluene. The ampoule was slowly warmed at about 30°C for one half hour and at about 80°C for the next two hours. During this period, it was necessary to freeze and evacuate the ampoule periodically every ten minutes to remove CO (a side product of the reaction). After this time period, the resulting solution contained in the ampoule was allowed to stay overnight at -10°C. Crystalline solid was filtered, washed with light petroleum ether and dried under vacuum. Yields and compound properties are listed below:

**Ni-1 [1,4-bis-(2,6-di-*iso*-propylphenyl)-1,4-diaza-1,3-butadiene] nickel(II)[3,6-di-*tert*-butylcatecholate].**  
Dark green air stable crystals. Yield 0.426 g (65%). IR (Nujol,  $\text{cm}^{-1}$ ): 1590 w, 1550 w, 1370 m, 1360 m, 1320 s, 1305 s, 1275 s, 1260 s, 1210 s, 1180 s, 1040 s, 985 s, 940 m, 870 m, 860 m, 785 s, 750 s, 700 s, 655 s, 620 m, 595 m, 515 m, 490 w. Anal.(%) Found: C 72.94; H 8.79; Ni 8.95.  $\text{C}_{40}\text{H}_{56}\text{N}_2\text{O}_2\text{Ni}$  Calc.: C 73.28; H 8.55; Ni 9.01. The complex is soluble in THF,  $\text{CH}_2\text{Cl}_2$  and  $\text{Et}_2\text{O}$ , is moderately soluble in toluene, and is insoluble in light petroleum ether

**Ni-2 [1,4-bis-(2,6-di-*iso*-propylphenyl)-1,4-diaza-1,3-butadiene] nickel(II)[3,6-di-*tert*-butyl-4,5-dichlorocatecholate].**

Dark green air stable crystals. Yield 0.47 g (65%). IR (Nujol,  $\text{cm}^{-1}$ ): 1590 w, 1565 w, 1545 w, 1400, 1370 s, 1330 s, 1305 m, 1260 s, 1240 s, 1220 s, 1180 s, 1035 s, 990 m, 865 m, 850 m, 800 m, 755 m, 700 m, 670, 600, 515. The complex is soluble in THF,  $\text{CH}_2\text{Cl}_2$ ,  $\text{Et}_2\text{O}$  and toluene, and is insoluble in light petroleum ether.

**Ni-3 [1,4-bis-(2,6-di-*iso*-propylphenyl)-1,4-diaza-1,3-butadiene] nickel(II)[3,6-di-*tert*-butyl-4,5-dimethoxycatecholate].**

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<i>B. Mitakach</i>	<i>J. McNeil</i>	
WITNESSED, READ & UNDERSTOOD BY	WITNESSED, READ & UNDERSTOOD BY	
<i>W. A. D.</i>	<i>R. A. S.</i>	
INVENTOR EMPLOYMENT CONTRACT	INVENTOR EMPLOYMENT CONTRACT	
ExxonMobil Chemical Co. DIVISION:	ExxonMobil Chemical Co. DIVISION:	
OTHER:	OTHER:	<i>Polymer Science</i>

Brown-green air stable crystals. Yield 0.5 g (70%). IR (Nujol,  $\text{cm}^{-1}$ ): 1560 w, 1530 w, 1500, 1395, 1370 s, 1330 m, 1285 s, 1230 s, 1180 s, 1150 m, 1115 m, 1090, 1045 s, 1025 m, 1005 m, 885 m, 870 m, 760, 730 s, 705 m, 690 m, 600 w, 575 w, 515 m. The complex is soluble in THF,  $\text{CH}_2\text{Cl}_2$ ,  $\text{Et}_2\text{O}$  and toluene, and is insoluble in light petroleum ether.

**Ni-4 [1,4-bis-(2,6-di-*iso*-propylphenyl)-1,4-diaza-1,3-butadiene] nickel(II)[3,5-di-*tert*-butylcatecholate].** Yellow-green crystals. Yield 0.265 g (40%). IR (Nujol,  $\text{cm}^{-1}$ ): 1590 m, 1530 m, 1475 vs, 1440 s, 1400, 1365 s, 1360 s, 1325 s, 1300 s, 1250 m, 1205 m, 1180 s, 1110, 1060 m, 1040 s, 1025, 990, 920, 870 m, 845, 830, 800, 760 m, 705, 660, 605, 525. The complex is soluble in THF,  $\text{CH}_2\text{Cl}_2$ , toluene, and is moderate soluble in light petroleum ether.

**Ni-5 [2,3-dimethyl-1,4-bis-(2,6-di-*iso*-propylphenyl)-1,4-diaza-1,3-butadiene] nickel(II)[3,6-di-*tert*-butylcatecholate].**

Dark green air stable crystals. Yield 0.546 g (80%). IR (Nujol,  $\text{cm}^{-1}$ ): 1595 w, 1580 w, 1505 s, 1405 s, 1370 m, 1345 s, 1320 s, 1305 s, 1275 s, 1215 s, 1065 w, 985 s, 950 m, 890 w, 880 w, 835 w, 785 m, 740 m, 715 w, 705 w, 655, 620 m, w, 515 w. The complex is soluble in THF,  $\text{CH}_2\text{Cl}_2$ ,  $\text{Et}_2\text{O}$  and toluene, and is insoluble in light petroleum ether.

**Ni-6 [1,4-bis-(2,6-dimethylphenyl)-1,4-diaza-1,3-butadiene]nickel(II) 3,6-di-*tert*-butyl- catecholate from [1,4-bis-(2,6-dimethylphenyl)-1,4-diaza-1,3-butadiene] nickel(II) dibromide.**

A solution of 1,4-bis-(2,6-dimethylphenyl)-1,4-diaza-1,3-butadiene (0.27 g, 1.0 mmol) in 20 ml THF was added to  $(\text{DME})\text{NiBr}_2$  (0.31 g, 1.0 mmol) suspended in 20 ml THF. The reaction mixture was warmed at about  $60^\circ\text{C}$  during one day until the precipitate disappeared. Deep-brown crystals were filtered, washed with cold light petroleum ether and dried in vacuum. Yield 0.29 g (60%). IR (Nujol,  $\text{cm}^{-1}$ ):  $\nu(\text{C}=\text{N})$  1630  $\text{cm}^{-1}$ .

To 0.48 g (1.0 mmol) of [1,4-bis-(2,6-dimethylphenyl)-1,4-diaza-1,3-butadiene] nickel(II) dibromide in 20 ml THF, a suspension of (3,6-di-*tert*-butyl-catecholate) di-thallium in 20 ml THF was added. The mixture was refluxed for one hour. The solvent was then changed to  $\text{CH}_2\text{Cl}_2$ /hexane (1/1). The solution was then filtered, reduced in volume and chilled for 10 hours. The resulting dark green solid was filtered, washed with cold hexane and dried in vacuum. Yield 0.30 g (55%). Anal. (%) Found: C 71.50; H 7.81; Ni 10.56.  $\text{C}_{32}\text{H}_{40}\text{N}_2\text{O}_2\text{Ni}$  Calc.: C 70.73; H 7.42; Ni 10.80. IR (Nujol,  $\text{cm}^{-1}$ ): 1720, 1600, 1555, 1460, 1360, 1320 s, 1305 s, 1245, 1205 s. The complex is air stable, and is soluble in THF,  $\text{CH}_2\text{Cl}_2$ , and is slightly soluble in toluene.

**Ni-7 [2,3-dimethyl-1,4-diphenyl-1,4-diaza-1,3-butadiene]nickel(II) 3,5-di-*tert*-butyl-catecholate)**

Nickel tetracarbonyl (0.86 g, 5 mmol) was condensed into an evacuated and frozen ampoule (having reserved volume of approximately 1 litre) containing 2,3-dimethyl-1,4-diphenyl-1,4-diaza-1,3-butadiene (1.11 g, 5 mmol) and 3,5-di-*tert*-butyl-o-benzoquinone (1.10 g, 5 mmol) in 100 ml of degassed toluene. The ampoule was slowly warmed at  $-30^\circ\text{C}$  for one hour and at  $-80^\circ\text{C}$  for the next two hours. Resulting solution was maintained at  $-10^\circ\text{C}$  overnight. Dark green crystals were filtered, washed with light petroleum and dried under vacuum. Yield 1.826 g (65%). IR (Nujol,  $\text{cm}^{-1}$ ): 1585, 1515 m, 1490 m, 1420, 1390, 1340 m, 1300 s, 1265 m, 1250, 1215, 1075, 985 s, 850 m, 830, 765 s, 730, 695 s, 655, 625, 525.  $^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): 0.94 and 1.11 s ( $2 \times 9\text{H}$ ,  $\text{C}(\text{CH}_3)_3$ ); 1.78 s (6H,  $\text{N}=\text{CCH}_3$ ); 7.27- 7.50 m (10H,  $2 \times \text{C}_6\text{H}_5$ ).

### Polymerizations

Polymerizations were conducted in a 1 L stainless steel Zipperclave reactor equipped with a paddle stirrer, a temperature controller, an on demand supply of ethylene regulated to maintain a constant reactor pressure,

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SIGNATURE (FIRST NAME IN FULL)	SIGNATURE (FIRST NAME IN FULL)	
<i>B. G. S. Squire</i>	<i>B. G. S. Squire</i>	
WITNESSED, READ & UNDERSTOOD BY	WITNESSED, READ & UNDERSTOOD BY	
<i>W. M. Squire</i>	<i>W. M. Squire</i>	
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ExxonMobil Chemical Co. DIVISION:	ExxonMobil Chemical Co. DIVISION:	
OTHER:	OTHER:	
	<i>Polymer Science</i>	

and a supply of dry high pressure nitrogen to maintain an inert atmosphere. Monomers and solvents directly plumbed into the reactor were passed through drying columns prior to entering the reactor.

A typical reaction to produce polyethylene consisted of adding 400 ml of dry toluene and the indicated amount of 10 wt% methylalumoxane in toluene (Albemarle) to the reactor. After addition, the reactor was vented to reduce excess nitrogen pressure. The reactor was heated to the desired reaction temperature and charged with the desired differential pressure of ethylene. The pre-catalyst dissolved in dry toluene contained in a catalyst addition tube was flushed into the reactor using approximately 20 ml of dry toluene under nitrogen pressure. The reaction was run for a period of 15 minutes at which time the ethylene flow was discontinued, and the reactor was quickly cooled and vented. The contents of the reactor were precipitated in methanol. The polymer was initially dried under a flow of nitrogen and then typically dried overnight in a vacuum oven at 90-100°C.

A typical reaction to produce polypropylene consisted of adding the indicated amount of dry toluene and 10 wt% methylalumoxane in toluene (Albemarle) to the reactor. After addition, the reactor was vented to reduce excess nitrogen pressure. Afterwards, the indicated amount of propylene was added to the reactor and the reactor was heated to the desired reaction temperature. The pre-catalyst dissolved in dry toluene contained in a catalyst addition tube was flushed into the reactor using approximately 20 ml of dry toluene under nitrogen pressure. The reaction was run for a period of 30 minutes at which time the reactor was quickly cooled and vented. The contents of the reactor were precipitated in methanol. The polymer was initially dried under a flow of nitrogen and then typically dried overnight in a vacuum oven at 90-100°C.

Molecular weights (weight average molecular weight ( $M_w$ ) and number average molecular weight ( $M_n$ )) were measured by Gel Permeation Chromatography using a Waters 150 Gel Permeation Chromatograph equipped with a differential refractive index detector and calibrated using polystyrene standards. Samples were run 1,2,4-trichlorobenzene (145°C) using three Shodex GPC AT-80 M/S columns in series. No column spreading corrections were employed but data on generally accepted standards, e.g. National Bureau of Standards Polyethylene 1475, demonstrated a precision with 0.1 units for  $M_w/M_n$ , which was calculated from elution times. Numerical analyses were performed using Expert Ease® software available from Waters Corporation.

Polymer comonomer incorporation was determined by  $^1\text{H}$  NMR using a Varian Unity+ 400 MHz instrument run with a single 30° flip angle RF pulse. A total of 120 pulses with a delay of 8 seconds between pulses were signal averaged. The polymer sample was dissolved in heated tetrachloroethane- $d_2$  and signal collection took place at 120°C.

The results of the polymerization experiments are tabulated below:

Table 1. Ethylene polymerization runs – part A

Run #	TMC	TMC Stock (mg)	Stock used (ml)	$\mu\text{mol}$ TMC used	10 wt% MAO (ml)	Al/M	Temp (C)	delta $\text{C}_2\text{H}_4$ (atm)	P (g)	kgP/molM $\cdot$ a tm $\cdot$ hr
22128-124-001	Ni-1	10	10	1.525	1.9	1246	80	4.422	0.8	468

INVENTOR(S)		DATE
SIGNATURE (FIRST NAME IN FULL) <i>mitakaur</i>	SIGNATURE (FIRST NAME IN FULL) <i>McConnell</i>	<i>11/11/01</i>
SIGNATURE (FIRST NAME IN FULL) <i>Berni Shaw</i>	SIGNATURE (FIRST NAME IN FULL) <i>KR Squire</i>	
WITNESSED, READ & UNDERSTOOD BY <i>W. H. H. H.</i>	WITNESSED, READ & UNDERSTOOD BY <i>[Signature]</i>	
INVENTOR EMPLOYMENT CONTRACT <i>[Signature]</i>	INVENTOR EMPLOYMENT CONTRACT <i>[Signature]</i>	
ExxonMobil Chemical Co. DIVISION: <i>[Signature]</i>	ExxonMobil Chemical Co. DIVISION: <i>Polymer Science</i>	
OTHER:	OTHER:	



22128-125-001	Ni-1	10	10	1.983	2.5	1261	80	4.422	1.0	456
22128-126-001	Ni-1	10	10	3.966	5	1261	80	4.422	1.9	433
22128-127-001	Ni-1	10	10	3.966	5	1261	80	4.422	1.4	315
22128-128-001	Ni-1	10	10	3.966	5	1261	60	3.537	9.5	2,697
22128-129-001	Ni-1	10	10	3.966	5	1261	60	3.537	10.6	3,031
22128-134-001	Ni-1	10	10	3.966	5	1261	40	2.721	7.4	2,747
22128-135-001	Ni-1	10	10	1.983	2.5	1261	40	2.721	16.9	12,528
22128-136-001	Ni-1	10	10	1.983	2.5	1261	40	2.721	18.2	13,507
22128-144-001	Ni-2	10	10	4.003	5	1249	80	4.422	2.2	499
22128-145-001	Ni-2	10	10	4.003	5	1249	80	4.422	2.2	499
22128-146-001	Ni-2	10	10	4.003	5	1249	80	4.422	2.3	529
22128-147-001	Ni-2	10	10	4.003	5	1249	60	3.537	8.3	2,350
22128-148-001	Ni-2	10	10	4.003	5	1249	60	3.537	8.6	2,415
22128-149-001	Ni-2	10	10	1.932	2.5	1294	40	2.721	20.3	15,465
22128-150-001	Ni-2	10	10	1.932	2.5	1294	40	2.721	21.3	16,218
22128-137-001	Ni-3	10	10	4.052	5	1234	80	4.422	1.9	429
22128-138-001	Ni-3	10	10	4.052	5	1234	80	4.422	0.0	0
22128-139-001	Ni-3	10	10	4.052	5	1234	80	4.422	1.9	431
22128-140-001	Ni-3	10	10	4.052	5	1234	60	3.537	9.2	2,573
22128-141-001	Ni-3	10	10	4.052	5	1234	60	3.537	11.7	3,262
22128-142-001	Ni-3	10	10	1.956	2.5	1278	40	2.721	20.5	15,404
22128-143-001	Ni-3	10	10	1.956	2.5	1278	40	2.721	20.8	15,659
21503-040-001	Ni-6	13.5	10	3.727	4.5	1207	80	4.422	1.3	311
21503-041-001	Ni-6	13.5	10	3.727	4.5	1207	80	4.422	0.6	143
21503-042-001	Ni-6	13.5	10	3.727	4.5	1207	60	4.422	0.5	131
21503-045-001	Ni-6	13.5	10	3.727	4.5	1207	60	4.422	0.7	158

Table 2. Ethylene polymerization runs – Part B

Run #	MW	Mn	MWD	SCB/ 1000C HNMR	Vinylenes /1000C	Trisubstituted /1000C	Vinyls /1000C	Vinylidenes /1000C
22128-124-001	44,476	20,515	2.17		0.31	0.00	0.21	0.20
22128-125-001	36,367	10,595	3.43		0.34	0.00	0.21	0.20
22128-126-001	24,661	10,043	2.46	51.2	0.68	0.01	0.63	0.05
22128-127-001	26,565	10,547	2.52	52.6	0.55	0.00	0.55	0.11
22128-128-001	78,175	26,136	2.99	30.4	0.14	0.01	0.33	0.06
22128-129-001	82,051	27,549	2.98	31.5	0.41	0.00	0.62	0.08
22128-134-001	87,552	26,881	3.26		0.31	0.00	0.21	0.20
22128-135-001	128,383	40,744	3.15	11.9	0.06	0.04	0.36	0.03
22128-136-001	126,189	42,484	2.97	12.3	0.04	0.00	0.29	0.00
22128-144-001	29,739	11,433	2.60		0.44	0.00	0.46	0.08
22128-145-001	38,998	16,310	2.39		0.51	0.01	0.50	0.05

INVENTOR(S)		DATE
SIGNATURE (FIRST NAME IN FULL) <i>Mitakaker</i>	SIGNATURE (FIRST NAME IN FULL) <i>Imcane</i>	<i>1/18/91</i>
SIGNATURE (FIRST NAME IN FULL) <i>Bruce Shue</i>	SIGNATURE (FIRST NAME IN FULL) <i>KR Squire</i>	
WITNESSED, READ & UNDERSTOOD BY <i>Walt Nater</i>	WITNESSED, READ & UNDERSTOOD BY <i>[Signature]</i>	
INVENTOR EMPLOYMENT CONTRACT	INVENTOR EMPLOYMENT CONTRACT	
ExxonMobil Chemical Co. DIVISION:	ExxonMobil Chemical Co. DIVISION:	
OTHER:	OTHER: <i>Polymer Science</i>	

22128-146-001	176,641	14,947	11.8*		0.36	0.00	0.40	0.07
22128-147-001	94,798	27,012	3.51		0.42	0.00	0.64	0.04
22128-148-001	82,891	24,447	3.39		0.21	0.00	0.31	0.04
22128-149-001	122,154	44,401	2.75		0.06	0.02	0.28	0.01
22128-150-001	125,810	37,279	3.38		0.08	0.02	0.29	0.01
22128-137-001	40,150	17,592	2.28	44.9	0.44	0.01	0.42	0.05
22128-138-001	-	-	-	-	-	-	-	-
22128-139-001	62,180	15,706	3.96	43.5	0.45	0.00	0.41	0.03
22128-140-001	75,871	24,424	3.11	32.5	0.22	0.04	0.37	0.03
22128-141-001	89,053	25,915	3.44	28.7	0.18	0.00	0.30	0.04
22128-142-001	117,747	40,699	2.89		0.06	0.00	0.30	0.01
22128-143-001	115,762	37,892	3.06		0.05	0.01	0.29	0.01
21503-040-001	622,221	250,223	2.49	5.6	0.17	0.19	0.13	0.44
21503-041-001	587,068	204,923	2.86	3.0	0.10	0.17	0.08	0.27
21503-042-001	678,423	323,291	2.10	6.8	0.10	0.07	0.08	0.63
21503-045-001	574,515	59,433	9.67	6.8	0.20	0.03	0.21	0.44

\*bimodal

Table 3. Propylene polymerization runs – Part A

Run #		TMC stock soln (mg)	Tol (ml)	μmol TMC used	1M MAO (ml)	Al/M	Tol (ml)	C <sub>3</sub> H <sub>6</sub> (ml)	Temp (C)	P (g)	kgP/mol TMC hr
21503-050-001	Ni-6	13.5	10	5.96	3	503	0	400	40	0.2	80
21503-051-001	Ni-6	14.5	10	6.40	3	468	0	400	40	0.2	72
22128-151-001	Ni-1	10.0	10	3.97	5	1261	250	250	40	2.4	1,230
22128-152-001	Ni-1	10.0	10	3.97	5	1261	250	250	40	2.8	1,432

Table 4. Propylene polymerization runs – Part B

Run #	MW	Mn	MWD	Mn (unsat)	Vinylenes /1000C	Trisubstituted /1000C	Vinyls /1000C	Vinylidenes /1000C
21503-050-001				1,691	2.52	2.43	1.35	1.98
21503-051-001				1,081	3.64	4.57	1.37	3.37
22128-151-001	118,966	68,576	1.74	53,846	0.08	0.03	0.09	0.06
22128-152-001	122,181	77,091	1.59	56,000	0.08	0.03	0.08	0.06

<b>INVENTOR(S)</b>	
SIGNATURE (FIRST NAME IN FULL) <i>milakacker</i>	SIGNATURE (FIRST NAME IN FULL) <i>DATE</i> <i>JM card</i> <i>KR Squire</i>
SIGNATURE (FIRST NAME IN FULL) <i>Bayer</i>	SIGNATURE (FIRST NAME IN FULL) <i>[Signature]</i>
WITNESSED, READ & UNDERSTOOD BY <i>[Signature]</i>	WITNESSED, READ & UNDERSTOOD BY <i>[Signature]</i>
INVENTOR EMPLOYMENT CONTRACT <input type="checkbox"/> Yes <input type="checkbox"/> No	INVENTOR EMPLOYMENT CONTRACT <input type="checkbox"/> Yes <input type="checkbox"/> No
ExxonMobil Chemical Co. DIVISION:	ExxonMobil Chemical Co. DIVISION: <i>Polymer Science</i>
OTHER:	OTHER: